ACCEPTANCE CRITERIA FOR SLIP-CAST FUSED SILICA RADOMES

Technical Report No. 3

June 1972

By

J. N. Harris
E. A. Welsh

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TABLE OF CONTENTS

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ABSTRACT

This report consists of an acceptance criteria specification for slip-cast fused silica radomes. The rationale behind the requirements for this specification is given.

I. PURPOSE

The purpose of Contract No. N00017-70-C-4438 is to perform research and development directed towards the development of techniques to fully exploit the potential of readily available ceramic systems for use as structural components in hypersonic missile applications.

II. INTRODUCTION

In order to determine whether a delivered radome or a radome blank meets particular specifications for the intended final use certain acceptance tests and measurements must be made. Material tests must be; either non-destructive, accomplished on material removed from an excess portion of the radome, or accomplished on separate material processed from start to finish at the same time and in the same manner as the radome. Acceptance tests should be simple to accomplish and should not be expensive or time consuming. The tests may be conducted by the supplier, the receiver, or jointly.

It must be remembered that acceptance tests are only a measure of the material under the specified test conditions and do not necessarily represent stress failure levels, etc. that may be experienced under actual flight conditions. The complete response (mechanical, electrical, and thermal) of a radome is intimately related to its particular mission and can only be determined by a detailed experimental and/or analytical study for each individual missile system.

To ensure that tests whether conducted by the supplier or the receiver, are standardized ASTM specifications have been cited wherever possible.

III. ACCEPTANCE TESTING REQUIREMENTS

A. Mechanical Properties

The strength of slip-cast fused silica increases with temperature to approximately 2000° F. Therefore, testing at room temperature represents a minimum strength and should be satisfactory for all acceptance tests.

1. Tensile Strength

In most radome shapes the simplest and easiest test specimen to prepare from the extended portion of the radome skirts is a machined ring. Several rings can be machined and subjected to a hydrostatic tensile test using equipment similar to that designed by Sedlacek 1/. At the same time the tensile test rings can be instrumented with strain gages to determine tensile elastic modulus and Poisson's ratio.

2. Modulus of Rupture

Depending on radome wall thickness and diameter, rectangular modulus of rupture specimens can be machined from the skirt section. If the diameter and wall thickness are sufficient, specimens can be cut from both the horizontal and vertical directions of the skirt section. Prior to breaking these specimens, modulus of elasticity can also be determined using a sonic resonance method as specified in ASTM C623 and/or by measuring the deflection of the modulus of rupture specimen as specified in ASTM E111. The modulus of rupture specimen is to be broken transversely, using four point loading to prevent wedging effects and to subject a larger volume of material to maximum stress.

B. Physical Properties

1. Density

Bulk density, porosity and water absorption can be determined on samples of sufficient size cut or broken from the skirt-section of the radome using the procedures of ASTM C373. In addition, determinations should be made on several samples taken from the base, middle and tip area of one radome from each lot.

2. Cristobalite Content

At least one bulk density sample from the skirt and one sample from each area of the test radome from each lot should be examined for bulk cristobalite content using x-ray diffraction comparison techniques as outlined in Appendix II.

3. Thermal Conductivity

The thermal conductivity of slip-cast fused silica is directly related to its porosity 2/, 3/. If the density and porosity are known, thermal conductivity can be estimated as close as the limits of experimental error in actual measurements. Therefore, there is no need to measure thermal conductivity on material from each radome.

4. Specific Heat

The specific heat does not vary with processing variations. Therefore, there is no need for determination of specific heat on each radome. Acceptable values for specific heat are given in the Fused Silica Design Manual 4/ published during this program.

5. Thermal Expansion

Thermal expansion should be determined on material from each radome using the procedures of ASTM E228.

C. Dielectric Properties

Dielectric constant and loss tangent should be measured from room temperature to 1000° C at the frequency(ies) to be used in service using the procedures of ASTM D2520.

D. Physical Dimensions

1. Wall Thickness

Wall thickness should be measured with a suitable device such as deep throated calipers or a specially constructed device such as that described by Poulos, et al., 5/. Sufficient measurements should be made vertically and circumferentially to be certain there are no variations in the wall thickness which exceed the prescribed tolerances.

2. Concentricity and Prescribed Contour

Concentricity should be measured using a suitable measuring technique to insure that the radome meets specifications. Out-of-roundness can occur in slip-cast fused silica radomes during slip casting and firing operations. Out-of-roundness should be removed during machining operations. However, if vacuum chucks are used, it is possible to machine a thin wall radome to a perfectly round condition and then have it go out of shape when the vacuum is removed. Radome contour should also be determined at several points to insure compliance with specifications

E. Cracks and Voids

The translucent nature of slip-cast fused silica allows it to be inspected for cracks and voids using a visual technique. By illuminating the interior with a 300 watt bulb and soaking the outside with isopropyl alcohol any cracks or voids become visible. Ten power magnification will aid in discovering small cracks or voids.

APPENDIX I

ACCEPTANCE TEST CRITERIA RADOME,
SLIP-CAST FUSED SILICA

1.0 Scope

This document outlines the procedures and equipment to be used in the Quality Acceptance testing of Radome Blanks as a demonstration of compliance to the performance requirements.

2.0 Applicable Documents and Drawings

ASTM C373

ASTM C674

ASTM D2520

ASTM E111

ASTM E132

ASTM E137

ASTM E228

Sedlaceck, R.; F. A. Halden; "Method for Testing Brittle Materials," Rev. Sci. Inscr., 33, (3), 298-300, 1962.

3.0 Test Conditions

Unless otherwise specified herein, or in the applicable test procedures, the acceptance testing of the Radome Blank will be performed at standard ambient conditions as defined below:

Temperature	25° C ± 10° C (77° F ± 18° F)
Relative Humidity	90 per cent Max with no Minimum
Barometric Pressure	29 to 32 inches of Hg
Test Equipment Warm-Up Time	2 hours Minimum

4.0 Test Equipment

Test equipment meeting the requirements of the documents listed in 2.0. shall be deemed satisfactory.

5.0 Preliminary Instructions

5.1 The test technician shall read and understand each test procedure prior to testing the Radome Blank, or associated test specimens.

5.1.1 Verify that the item is the correct serial number, and proper configuration.

5.1.2 Verify that test instruments used are within calibration dates stated on calibration tag, or are properly calibrated where such calibration is necessary prior to each use.

5.1.3 Record appropriate calibration dates and/or data on all applicable test data sheets.

5.1.4 Record all test results on the applicable data sheets.

5.2 All test results shall be within the limits specified on the applicable data sheets.

6.0 Acceptance Inspection Tests

6.1 The following paragraphs detail procedures for demonstrating performance requirements for material to be used in radome

manufacture. All measurements with the exception of chemical analysis are to be made on suitable test specimens processed under the same conditions as deliverable radomes.

6.1.1 Composition. Chemical analysis conforming with ASTM E137 shall be furnished by the material vendor. Cristobalite content shall be determined by X-ray diffraction, as specified by Georgia Tech method "Procedure for Determining Bulk α-Cristobalite Content by X-ray Diffraction Techniques."

6.1.2 Bulk Density. Bulk density shall be determined from a minimum of five (5) test samples per ASTM C373.

6.1.3 Modulus of Rupture. A minimum of three (3) test specimens shall be diamond ground from a suitable slip-cast fused billet, using distilled water as a coolant. Samples will be broken according to ASTM C674, with the exception that quarter point loading shall be used.

6.1.4 Tensile Strength. Tensile strength shall be determined by a hydrostatic test on a minimum of two (2) rings machined from the skirt section of the radome blank. Testing shall be performed with suitably sized equipment and with procedures conforming to the recommendations of Sedlacek and Halden.

6.1.5 Poisson's Ratio. Poisson's Ratio shall be measured utilizing strain gages attached to the hoop tensile specimen.

6.1.6 Modulus of Elasticity. Young's Modulus shall be determined at room temperature utilizing strain gages on the hoop-tensile specimen.

6.1.7 Dielectric Properties. Dielectric constant and loss tangent shall be measured at the frequency(ies) to be used in service from 20° to 1000° C in accordance with ASTM D2520.

6.1.8 Thermal Expansion. Thermal expansion shall be determined per ASTM E228 at temperatures from 20° to 1000° C.

6.2 The following paragraphs detail procedures for demonstrating compliance with the Quality Conformance Inspection.

6.2.1 Wall Thickness. Wall thickness shall be measured at a minimum of 20 points spaced 90 degrees apart around the circumference of the radome.

6.2.2 Cracks and Voids. The radome shall be soaked with isopropyl alcohol, illuminated from within with a 300 watt light bulb, and inspected visually at 10X magnification for the presence of cracks and voids.

6.2.3 External Dimensions. The external dimensions shall be measured with either pi tape, or engraved steel rule to the maximum accuracy obtainable with the particular measuring device.

6.2.4 Roundness. Roundness measurements will be made using a suitable rotational device.

APPENDIX II

PROCEDURE FOR DETERMINING BULK α-CRISTOBALITE CONTENTS BY X-RAY DIFFRACTION TECHNIQUES

This procedure is presented to serve as a guide to installations which desire to utilize this technique for the determination of α-cristobalite in slip-cast fused silica.

α-Cristobalite Standard

Since no absolute X-ray standard exists for α-cristobalite, the many x-ray diffraction studies of α-cristobalite have always utilized an α-cristobalite reference or standard prepared from some other form of silicon dioxide. The starting materials in these preparations have ranged from well crystallized quartz to silicic acid. According to the work of Floerke 6/, the quantitative analysis of α-cristobalite by x-ray diffraction requires special care to relate the calibration or reference material to the estimated α-cristobalite phase. This requirement was further emphasized by the experimental studies of Foster et al. 7/; the starting form of silicon dioxide which produces the α-cristobalite standard should be made of material similar to that being analyzed. Also, the standard should be produced at a temperature level at which the specimens to be analyzed will be devitrified. The following procedure describes preparation of one standard.

The α-cristobalite standard was prepared from fused silicon dioxide obtained by the fusion of quartz sand in an electric arc. The material is typical of that used in the fabrication of slip-cast fused silicon dioxide bodies. A slip-cast cylindrical specimen was sintered for 60 hours at 2500° F in an electric furnace with an air atmosphere. The specimen was subsequently crushed and ground to minus 325 mesh powder.

Sizing and Packing

Test samples for α-cristobalite content determinations are crushed and screened through a 325 mesh screen to preclude preferred orientation due to overly large particles. The samples are packed against a frosted glass slide to help maintain random orientation of the powder and provide for a reproducible "roughness" of the sample surface. Repacking studies have shown that when this procedure is used the variations from sample to sample due to preferred orientation and to surface roughness are quite small. In addition, no variation in diffracted beam intensity with packing density has been observed 8/.

Scanning

Scanning from 21 to 22.67 degrees two-theta (2θ) is sufficient for the measurement of the (101) α-cristobalite reflection (2θ = 21.94 degrees).

Basis

The integrated intensity is used, since it is relatively insensitive to the crystallite sizes and the residual inhomogeneous strains in the crystals. The integrated intensity is a most convenient index since it has been found to be directly proportional to the percentage of the standard prepared by mixing known quantities of standard and fused silicon dioxide over the range 0.5 to 20.0 per cent of the standard cristobalite sample. The overlapping of $K\alpha_1$ and $K\alpha_2$ lines is avoided when integrated intensities are used.

Data duction

Diffractometer traces are made on both the standard and the test sample. A background curve can be established to eliminate those portions of the

spectra which do not constitute the α-cristobalite (101) reflection intensity. The α-cristobalite (101) reflection occurs atop a broad "hump" which is characteristic of fused silicon dioxide. Subjective judgment is involved in the placement of the background curve for low α-cristobalite contents. The subjectivity can be limited by background curve templates which fit the fused 100 per cent silicon dioxide hump in the traces for various incident intensities and scale factors. The traces from the standard and the sample can be measured with a planimeter to obtain integrated intensity data. Graphical representation of the integrated intensity is the area bounded by the diffraction trace and the background curve, expressed as the per cent "apparent α-cristobalite content" for the standard. For known amounts of standard mixed with fused silicon dioxide the apparent α-cristobalite content was found to be reproducible for measurements to about 20 per cent.

A second technique to limit the subjectivity of placing a background curve is to use an "artificial background curve" in the form of a straight line segment. In place of the conformal background curve, a straight line segment joining two convenient points on the trace is used. The points on the diffraction traces corresponding to a 2θ angle of 21.00 degrees and 22.67 degrees have been selected, and the apparent α-cristobalite content is simply based on the areas bounded by the diffraction traces and the straight line segments.

The technique has the advantage of area measurement under the diffraction peak from the integrated-count data which most x-ray units provide. For proper utilization of the integrated-count technique a correction method must be developed which allows separation of the diffracted energy associated

with the amorphous hump. Such a correction method was developed using mechanical mixtures of fused silicon dioxide and the cristobalite standard. The "apparent α-cristobalite content" of each of these mixtures was the integrated count for each mixture expressed as a percentage of the integrated count for the cristobalite standard. These apparent α-cristobalite contents were plotted as a function of the known content of cristobalite for the mixtures. This plot is used to estimate the α-cristobalite content of any test specimen from the integrated counts on the specimen and the cristobalite standard.

It must be emphasized, that the correction curve is not independent of the materials, procedures and equipment employed. Other laboratories must establish such a correction curve using the materials, procedures, and equipment which they employ.

Analysis Procedure

The following machine settings are used:

Tube: Cu

Filter: Ni

Tube Voltage: 40KV

Tube Current: 20 ma

Time Constant: 0.5

Scanning Rate: 2°/min

Divergent Slit: 1°

Receiving Slit: 0.003"

Scatter Slit: 1°

Diff-Int Switch: Diff.

The diffracted beam intensity from the cristobalite standard is amplified to a maximum by adjusting the Detector #2 Fine Voltage setting with the goniometer set at 21.94 degrees. The following procedure is used to obtain the α-cristobalite content of a sample, relative to the cristobalite standard.

1. With the unknown sample in place the goniometer is set at 21.00 degrees, and the count for a fixed time of 10 seconds is recorded.

2. The goniometer is operated at 2 degrees/min scanning rate, and the count for a fixed time of 50 seconds is recorded (total scan of 1.67 degrees two-theta).

3. At the end on the 50 second run of step 2 the goniometer setting is 22.67 degrees. At this setting the count for a fixed time of 10 seconds is recorded.

4. A "corrected count" is obtained by adding the two counts from steps 1 and 3, multiplying by 2.5, and subtracting from the number obtained in step 2. Graphically this corrected count represents the area bounded by the diffraction trace and the straight line segment for the test sample.

5. The "corrected count" for the cristobalite standard, which represents the area bounded by the diffraction trace and the straight line segment for the cristobalite standard, is obtained by repeating steps 1 through 4 with a cristobalite standard specimen.

6. The apparent α-cristobalite content is obtained by multiplying the corrected count for the sample by 100 and dividing by the corrected count for the cristobalite standard.

7. The α-cristobalite content, relative to the cristobalite standard, is obtained by reading the corrected value corresponding to the apparent α-cristobalite content obtained in step 6.

Sample Calculation

For a typical sample, the 10 second and end-counts at 21.00 and 22.67 degrees were 76 and 70, respectively; the 50 second count accumulated between 21.00 and 22.67 degrees was 460. For the cristobalite standard, the respective counts were 55, 45, and 1364. Thus:

The corrected count, test sample = 460 - 2.5 (76 + 70) = 95;

The corrected count, cristobalite standard = 1364 = 2.5 (55 + 47) = 1109;

The apparent α-cristobalite content = $\frac{95}{1109}$ x 100 = 8.6 v/o; and

The α-cristobalite content, relative to the standard as 100 per cent = 7.7 v/o.

Reporting Results

It has been the practice to report the interval expected to contain the α-cristobalite content of a test material at the 95 per cent confidence level which requires multiple determinations on a given sample; and, generally, two determinations are made on the A-4 standard for comparison.

Confidence Limits for α-Cristobalite Determinations 9/

Suppose n diffraction measurements are made on a given sample and two diffraction measurements are performed on the cristobalite standard, one

before and one after the measurements on the sample being analyzed. Let

$$A_i = \text{corrected count, cristobalite standard, } i = 1,2 \tag{1}$$

$$X_i = \text{corrected count, test sample, } i = 1,2,---,n \tag{2}$$

Then the sample variances would be

$$S_x^2 = \frac{n \sum_{i=1}^{n} x_i^2 - \left(\sum_{i=1}^{n} x_i\right)^2}{n\,(n-1)}, \tag{3}$$

and

$$S_A^2 = \frac{2 \sum_{i=1}^{2} A_i^2 - \left(\sum_{i=1}^{2} A_i\right)^2}{2} \tag{4}$$

for the sample count and the cristobalite standard count, respectively. The apparent α-cristobalite content, C, in the sample would be

$$\frac{\bar{X} - \frac{S_x}{\sqrt{n}} t_{n-1,\,\alpha}}{\bar{A} + \frac{S_A}{\sqrt{2}} t_{n-1,\beta}} \times 100 \le C \le \frac{\bar{X} + \frac{S_x}{\sqrt{n}} t_{n-1,\alpha}}{\bar{A} - \frac{S_A}{\sqrt{2}} t_{1,\beta}} \times 100, \text{ with 95\% confidence} \tag{5}$$

where $\alpha\beta = 0.025$ and

$$\bar{X} = \frac{1}{n} \sum_{i=1}^{n} X_i , \tag{6}$$

and

$$\bar{A} = \frac{1}{2} \sum_{i=1}^{n} A_i \, . \tag{7}$$

It is conservative to choose $\alpha = \beta = 0.158$ so that:

$$\frac{\bar{X} - \frac{S_x}{\sqrt{n}} t_{n-1,0.158}}{\bar{A} + \frac{S_A}{\sqrt{2}}(1.87)} \times 100 \leq C \leq \frac{\bar{X} + \frac{S_x}{\sqrt{n}} t_{n-1,\,0.158}}{\bar{A} - \frac{S_A}{\sqrt{2}}(1.87)} \times 100, \text{ with 95\% confidence} \tag{8}$$

The values of $t_{n-1,0.158}$ appear below:

n	$t_{n-1,0.158}$
2	1.87
3	1.02
4	0.93
5	0.91

Computer Program

To reduce the mathematical exercise required by the statistical calculations for a 95 per cent confidence interval, a computer program was prepared which contains a best fit curve for the correction factor to be used for α-cristobalite determination.

Language - ALGOL

N = number of measurement/sample

A_1, A_4 = 10 second count at 21.00 degrees for A-4 standard

A_2, A_5 = 50 second scan count from 21.00 to 22.67 degrees for A-4 standard

A_3, A_6 = 10 second count at 22.67 degrees for A-4 standard

X_1, X_4 = 10 second count at 21.00 degrees for unknown

X_2, X_5 = 50 second scan count from 21.00 to 22.67 degrees for unknown

X_3, X_6 = 10 second count at 22.67 degrees for unknown

N = sample code number

```
BEGIN
COMMENT         CRISTOBALITE CONTENT N=2;
INTEGER         N,A1,A2,A3,A4,A5,A6,X1,X2,X3,X4,X5,X6;
REAL            A,X,B,Y,Li,L2,L, H1,H2,H,E1,E,F1,F2,F3,F4,F5,F6,
                G1,G2,G3,L4,H4,E4,A7,A8,X7,X8;
FILE IN         PBIN (2,10);
FILE OUT        PBOUT  6 (2,15);
FORMAT OUT      FT1(X10,"CRISTOBALITE CONTENT N-2"///);
FORMAT OUT      FT2(X4,I6,X8,F7.2,X6, "TO", F9,2,X10,F7.2,/);
FORMAT OUT      FT3(X8,"SAMPLE",X8,"PER CENT CRISTOBALITE",.
                X9,"CODE",X9," (NINETY-FIVE PER CENT",X10,
                "EXPECTED",/,S8,"NUMBER",X8, "CONFIDENCE"
                "INTERVAL)",X11, "VALUE",/,/);
```

```
LABEL          BIGDADDY,FINISHED;
               WRITE (PBOUT,FT1);
               WRITE (PBOUT,FT3);
BIGDADDY:      READ (PBIN,/,A1,A2,A3,A4,A5,A6,N,X1,X2,X3,X4,X5,
               X6)[FINISHED];
               A7←A2-(2.5)xA1-(2.5)xA3;
               A8←A5-(2.5)xA4-(2.5)xA6;
               X7←X2-(2.5)xX1-(2.5)xX3;
               X8←X5-(2.5)xX4-(2.5)xX6;
               A←(0.5) x A7 + (0.5)xA8;
               X←(0.5) x X7 + (0.5)xX8;
               B←ABS(A7 - A8);
               Y←ABS(X7 - X8);
               L1←(100) x X - (93.3) x Y ;
               L2←A + (0.933) x B ;
               L←L1 / L2 ;
               H1←(100) x X + (93.3) x Y ;
               H2←A - (0.933) x B ;
               H←H1 / H2 ;
               E1←(100) x X ;
               F1←ABS(L - 7.5);
               F2←ABS(H - 7.5);
               F3←ABS(E - 7.5);
               F4←SQRT(F1);
               F5←SQRT(F2);
               F6←SQRT(F3);
```

```
        G1←(34.2) / (F4 + 7);

        G2←(34.2) / (F5 + 7);

        G3←(34.2) / (F6 + 7);

        L4←L-G1-(0.0097)xL+3;

        H4←H-G2-(0.0097)xH+3;

        E4←E-G3-(0.0097)xE+3;

        WRITE (PBOUT,FT2,N,L4,H4,E4);

        GO TO BIGDADDY:

FINISHED:  END.
```

CRISTOBALITE CONTENT N=2

SAMPLE CODE NUMBER	PER CENT CRISTOBALITE (NINETY-FIVE PER CENT CONFIDENCE INTERVAL)			EXPECTED VALUE
1	1.66	TO	2.34	1.99
3	0.95	TO	1.09	1.02
4	3.37	TO	3.61	3.49

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